

White Paint Analysis

George H. Ellis

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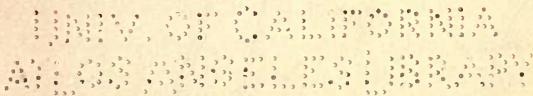


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Analysis of White Paints

A COLLECTION OF NOTES ON
THE CHEMICAL ANALYSIS
OF WHITE AND TINTED PAINTS



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... Late Chemist ...

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PREFACE.

THE following pages appeared originally in the columns of the "Paint, Oil and Drug Review," and they are now offered in book form at the request of chemists and many others in the paint trade.

While there has been much literature published in the technical and trade journals, yet there is no volume which treats solely of the chemical analysis of paints, and it is hoped that this little book may at least partially fill the place.

The author wishes to here express his obligations for valuable advice and the many suggestions offered by Mr. Walter Lee Brown, Prof. J. H. Long, Mr. Roger S. Pitkin, and particularly to Prof. Vernan J. Hall for his careful revision of proof.

G. H. E.

Chemical Laboratory,
May, 1899.

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INTRODUCTORY REMARKS.

My object in sending forth the following pages is to place in the hands of chemists and those interested in paints and pigments, a guide for the *practical* analysis of such products. I make no pretensions to having placed herein much of original matter, but have drawn freely from the records of chemical literature and authorities on the subject, to which I have added such other matter as my experience as an analytical chemist would warrant.

My aim has been to detail methods for the analysis of the majority of white and tinted pigments and pigment mixtures found in the paint trade, and which the chemist may be called upon to examine.

It is assumed that the chemist is familiar with the apparatus, reagents, etc., that are required in analytical chemistry, and for this reason their descriptions are omitted.

It has been my endeavor to give such methods and in sufficient detail that those having but a limited knowledge of the principles of analytical chemistry could successfully carry out the analyses. In this class there may be some in the paint trade who have sufficient knowledge of analytical chemistry to perform analyses and obtain results for their own use. To these I would respectfully suggest that the details of each determination be thoroughly studied in the standard works, such as Fresenius, in the hope that such research and practice may lead to that proficiency which belongs to the technical chemist.

For the guidance of those having but a limited experience the following general precautions are given:

1st. Generally the reagents used should be those known as chemically pure (c. p.) Should the standard chemicals of known purity be not at hand then the reagent should be especially tested and if found impure be rejected. Or if desired, it may be purified.

2nd. Use reagents intelligently. Too much as well as too little may cause errors. Study the reaction and the successful conditions required.

3rd. All distilled water should be tested for mineral salts. This may be done by evaporating several litres to dryness and should leave no perceptible residue.

4th. The amount of sample taken for analysis should be governed by the amount present of the constituent sought. In general one gram is a sufficient amount, but often times, if the constituent be but a trace, five times or more may be necessary to get a sufficient weighing amount. And again it may be that only a fraction of a gram will give a precipitate of sufficient size for easy manipulation.

5th. If a determination requires a special apparatus, first make a blank test or test with a substance containing the constituents of known amount, to make sure that the apparatus is in perfect working order. In fact all determinations may be practiced, using substances of known percentages.

6th. In using filter paper the size 9 c. m., S. and S. is to be preferred, requiring no correction, as they are practically ashless. If cheaper grades be used, always deduct the ash content. Little time is saved by using large filters as they require more washing and thereby increase the volume of the filtrate. The Gooch crucible and cone are recommended whenever admissable.

CHAPTER I.

PREPARING SAMPLES FOR ANALYSIS.

If the sample is in the form of a mixed or paste paint it will be advantageous to first free the pigments from the mixing fluid. This may be done in several ways.

When only the analysis of the pigments is desired the simplest method is to wash out or exhaust the oil and thinness by benzine, gasoline, petroleum-ether, benzole, ether, etc. As benzine or gasoline are the cheapest they are to be preferred. About 25 grams of the sample, previously *thoroughly* stirred, are placed in a beaker of about 150 c. c. capacity (or a whisky glass with thin sides) and after adding the solvent, the mass is stirred with a glass rod and the pigment allowed to settle. When the liquid is perfectly clear draw off the clear portion by means of a pipette, again add the solvent, stir and allow to settle. Usually three or four applications of the solvent will be found sufficient to free the pigment from oil and most of the drier. Any traces of the latter are disregarded. It is a good plan to test the solvent after the second treatment by evaporating a small portion on a watch crystal and observing whether it leaves any residue of oil. When ether is not used it is a good plan to finish the extraction with it. After drawing off the solvent as closely as possible the remainder is evaporated on a water bath and the residue heated in an air oven at 100° C. for about an hour. Remove the dry pigment to a glass or agate mortar, rub up thoroughly and pass through a fine sieve. Place in a well-stoppered bottle and label.

Should the sample be dry or not mixed with oil it is best to heat in the air oven at 100° C. in order to free it from moisture. Then place in bottle and keep stoppered.

If it is desired to ascertain the *percentage* of pigments and mixing fluid it may be done by weighing the beaker and

rod, then weighing again after adding the sample, the increase in weight being the amount of sample. Then, after exhausting and drying, the difference in weight is the amount of mixing fluid.

Example:—

Weight of beaker rod and sample.....	42.0771	grams.
Weight of beaker and rod.....	28.6435	"
Weight of sample.....	13.4336	"

After treating with solvent:—

Weight of beaker, rod and sample.....	42.0771	grams.
Weight of beaker, rod and pigments.....	36.0520	"
Loss equals mixing fluid.....	6.0251	"

then $\frac{6.0251 \times 100}{13.4336} = 44.858$ = percentage mixing fluid by weight.

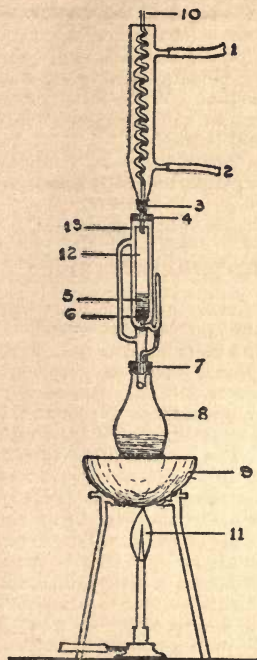
When it is necessary to be extremely accurate in getting both the percentages of mixing fluid and pigment it will be found best to exhaust the sample in a Soxhlet extractor, using petroleum-ether or ordinary sulphuric ether as a solvent. A convenient size of Soxhlet extractor is one inch diameter and nine inches in length with the arrangement as shown in the cut on page 5. The inside tube is an ordinary test tube with a hole cut or blown in the bottom. The bottom of the tube is then packed with a piece of cotton (6). The tube (12) and cotton are weighed first, then the sample of the paint is added, and the whole weighed again, the increase being the weight of sample taken. Another plug of cotton will be found necessary in cases when the pigment is so light that it floats to the top of the test tube. This occurs usually when the cotton is packed too tightly to permit the solvent to flow out of the bottom and often times it will be necessary to place a small glass stopper on the upper layer as a weight to prevent the contents of the tube from overflowing or floating to the top. When such an arrangement is necessary the whole tube, including sample and two plugs of cotton, are weighed, but not the glass which was added as a weight. About an ounce of the solvent, ether or petroleum-ether, is then placed in the flask and the water heated in the water bath not quite to boiling point. Soon the ether will begin to appear in the

condenser and drop back in the test tube containing the paint,

and, passing down through the sample, will fill up in the tube (13) and syphon over. This operation is kept up until the solvent liquid is perfectly clear and shows no color, which, in most cases, would be due to oil. Usually about a dozen syphonings will be sufficient, but samples vary and it is best to allow the extractor to work until the solvent syphons perfectly colorless. The test tube (12) containing the oil-free paint is now removed and thoroughly dried in a drying-oven then weighed.

The loss in weight equals the oil present, and the dried sample is taken out to be used for the analysis. If it is desired to find the amount of oil, the small flask (8) may be weighed before the operation and then the ether evaporated on a water bath heated in an air oven at 100° C, for 15 minutes, and the amount of the extracted oil weighed.

The centrifugal machine, such as is used in the separation of fat in milk, may be utilized to advantage in separating the pigment from the oil and thinners. This is accomplished by using from five to ten grams of the sample, and, after placing in the tube, add gasoline to within a half inch from the top. Then with a glass rod stir the whole until the



- | | |
|------------------|-------------------|
| 1 Water Outlet | 8 Glass Flask |
| 2 Water Inlet | 9 Water Bath |
| 3 Rubber Stopper | 10 Condenser Worm |
| 4 Cork Stopper | 11 Gas Flame |
| 5 Sample Paint | 12 Test Tube |
| 6 Cotton Plug | 13 Soxhlet Tube |
| 7 Cork Stopper | |

sample is thoroughly mixed with the benzine. Place in

machine and revolve about ten minutes or until the benzine is clear and free from the suspended pigment. Pour off the benzine, which is readily done as the pigment will be thrown to the bottom of the tube and forms a hard cake. Now add more benzine, and, with the aid of a glass rod, again stir thoroughly and revolve as before. Generally three or four treatments will complete the extraction. When the pigment is removed with the aid of a spatula and transferred to a watch glass. Then dried, sifted and bottled.

This method may be found of much value in a laboratory where many ground or mixed paints are daily examined and quick results desired.

It is the practice of some paint chemists to treat the sample, if it be mixed with oil, with strong nitric acid, to which is added about one-fourth water, and boil. The oil collects in a gummy mass and is filtered after diluting. Wash the filter with hot water acidulated with nitric acid. The filtrate will contain soluble pigments and the insoluble portion will remain on filter with the oil.

This method is not to be recommended except in cases where a rough test is desired to ascertain the bases present.

CHAPTER II.

WHITE PIGMENTS.

1. **Calcium Carbonate, (*Whiting*).**—Whiting consists of carbonate of calcium (CaCO_3), with small amounts of magnesium carbonate, silica, iron and alumina, moisture and possibly sulphate of calcium. The analysis is not often required as its cheapness offers no inducement for adulteration, yet the paint manufacturer or chemist may be called upon to choose between several samples and a plan for its examination is herewith given.

Moisture.—Take about two grams on a watch glass, weigh carefully, place in an air oven, and heat at a temperature of 100°C. , until the weight is constant. The loss is the moisture.

Silica and Insoluble Matter.—Dissolve one gram of the dried sample in a 200 c.c. covered beaker with about 10 c.c. strong hydrochloric acid and 100 c.c. of water. When effervescence is over and a white insoluble residue appears, it indicates that silica is present. Transfer to a platinum dish and bring to dryness on a water bath, then heat in an air oven at 100°C. , to drive off all the water and and render the silica insoluble. When the smell of hydrochloric acid has disappeared, remove from oven and moisten with hydrochloric acid, then add about 30 c.c. water and heat until everything but silica is dissolved. This will settle to the bottom and is filtered off on a small ashless filter and washed with hot water. Allow to drain well in funnel, place filter in a weighed platinum crucible and ignite until nothing but the white silica (SiO_2) remains. In igniting a wet or moist precipitate a little skill is required to prevent loss from spirting. If the operator will be careful to incline the crucible on the triangle, and allow the gas flame to play on the outer edge first, and gradually heat the whole crucible until the paper becomes charred,

forming a sort of envelope with the precipitate inside, no trouble will be occasioned from loss and the paper will gradually burn away. The precipitate is then cooled in the dessicator and weighed.

Iron and Alumina.—To the filtrate add a few drops of nitric acid, warm to oxidize the iron, then add 5 grams chemically pure ammonium chloride, then ammonia in excess and boil until excess of ammonia is driven off. Filter, wash with hot water and ignite the wet precipitate as in silica determination. Should a white flocculent precipitate appear, it shows that enough ammonium chloride has not been used to prevent the calcium from precipitating as hydrate, and it will be necessary to keep adding ammonium chloride until nothing but iron and alumina are thrown down. Weight equals iron oxide and alumina (Fe_2O_3 and Al_2O_3). As the amount of iron and alumina is very small in whiting, it will not be necessary to separate them.

Calcium Carbonate.—To the filtrate from the iron and alumina add ammonia in excess. Now add drop by drop and with constant stirring, ammonium oxalate (about 40 c.c. saturated solution) until the precipitate ceases to form and the ammonium oxalate is in considerable excess. It is necessary to add a sufficient amount to combine with the magnesium as well as the calcium. Allow the precipitate to settle in a warm place for several hours, preferably over night. Then pour the clear supernatant fluid through an ashless filter until the precipitate is covered with about a half inch of the liquid. Add hydrochloric acid until the precipitate is dissolved, then 40 c.c. hot water, ammonia in excess, add 5 c.c. ammonia oxalate, stir well and allow to stand again for several hours and filter. The filter is washed well with hot water and then dried. The object in dissolving and reprecipitating the calcium oxalate is to make sure that no magnesium is precipitated. In whiting it is not absolutely necessary, as the magnesium, if any is in small amounts, but in cases where much magnesium is present, it is quite essential for complete precipitation. Proceed with the filtrate as directed in magnesium determination.

The dried calcium oxalate may be weighed either as calcium oxide or calcium sulphate. If the former method is

to be used the ordinary heat of a Bunsen burner will not be sufficient to drive off all the carbon dioxide, and it will be necessary to heat over a blast lamp until after several weighings, the weight is constant. The weight of calcium oxide (CaO) multiplied by 1.784 gives the CaCO_3 or carbonate of calcium. If the calcium oxalate is to be converted to calcium sulphate proceed as follows: Moisten the dried precipitate of calcium oxalate with dilute sulphuric acid (1 part acid, 3 parts water) and heat cautiously until the excess of acid is driven off and the precipitate is white. Cool. Add a few drops of concentrated sulphuric acid and again heat cautiously by allowing the gas flame to play gently back and forth on the crucible until the white fumes are entirely driven off. Next heat to redness for about five minutes, cool and weigh. The weight of calcium sulphate multiplied by .7349 gives the CaCO_3 .

Magnesium Carbonate.—To the combined filtrates from the calcium oxalate precipitate add a large excess of ammonia until it is strongly alkaline, then an excess of a solution of sodium phosphate, stir well without permitting the rod to touch the sides of the beaker, allow to stand 12 hours (best over night) in a cool place, filter and wash with ammonia water (1 part ammonia, 2 parts water.) If the combined filtrate exceeds 100 c.c. add hydrochloric acid in slight excess, evaporate to 100 c.c., cool and proceed as above. A rubber tipper rod will be found very useful to rub the crystalline precipitate from the side of the beaker. Dry. Remove precipitate from paper with a spatula to a piece of black glazed paper, then burn the paper first in a weighed porcelain crucible, cool, add precipitate and ignite strongly until the precipitate is white or nearly so, and has a constant weight. Weigh the magnesium pyrophosphate ($\text{Mg}_2\text{P}_2\text{O}_7$) and multiply by factor .7575 to convert to MgCO_3 .

Sulphate.—To estimate any sulphuric acid that might be present as sulphates proceed as follows: 5 grams of the sample are dissolved in hydrochloric acid and any insoluble matter filtered off and the filter washed with hot water. To the warm filtrate, add drop by drop with constant stirring, a solution of barium chloride until the precipitate ceases to form and the precipitant is in excess. Allow to stand in a

warm place over night, filter, wash with hot water and after the precipitate is drained well, ignite as in silica determination. Weigh the white precipitate of barium sulphate (BaSO_4) and multiply by factor .583 to convert to calcium sulphate (CaSO_4) Then multiply the calcium sulphate found by factor .735 to convert to calcium carbonate, subtract this from the total calcium carbonate and the difference is the amount of calcium carbonate.

2. Gypsum (*Sulphate of Calcium, Terra Alba*).—Gypsum is found quite abundantly and often in a state of great purity. It has the chemical composition of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, and if the water be driven off by heat the well known Plaster of Paris will be left, which has the property of again combining with water to "set" into a white hard mass. When used as a pigment gypsum is usually fully hydrated.

Its analysis is not attended with any difficulty. The determinations ordinarily to be made are: insoluble matter, (such as clay, sand, etc.,) calcium, sulphuric acid, water and any small amounts of iron and alumina or magnesium. Proceed as directed on page 7 in the analysis of whiting for the determinations of soluble matter, iron and alumina, calcium and magnesium, and sulphuric acid. To determine water, use one gram and ignite in a platinum crucible to constant weight. For sulphuric acid determination use 1 gram instead of 5 as in whiting.

The bases and acids in gypsum are usually reported without combining them:

ANALYSES OF NATURAL GYPSUM BY THE AUTHOR.

	No. 1.	No. 2.
Calcium Oxide	32.98	32.32
Sulphuric Acid	45.79	45.76
Water	20.98	21.39
Silica	undet	.07
Iron Oxide	undet	.01
	99.65	99.55

3. China Clay (*Kaolin*).—China clay is a natural product and is essentially a hydrated silicate of alumina of the approximate composition, silica 47 per cent., alumina 40 per cent. and water 18 per cent., and has a formula $2\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$. It is not, however, of constant composition as clays from

different localities vary and usually contain small quantities of iron oxide with calcium, magnesium, potassium and sodium silicates.

The following is the method of analysis:

The sample after being pulverized and dried at 100°C ., for about an hour is kept in a well stoppered bottle. As clay is only slightly acted on by acids, it will be necessary to fuse it to effect decomposition.

Proceed as follows:

Silica.—One gram is thoroughly mixed in an agate mortar with 10 grams sodium carbonate and about a half a gram of potassium nitrate and placed in a capacious platinum crucible and covered. The mixture should not fill the crucible more than half full. Fuse over the blast lamp, run it carefully up the sides of the crucible, and when the fused mass is quite clear and quiet, allow to cool. Place the crucible and cover in a beaker, add water until the crucible is just covered and digest on a hot plate until the fused mass is thoroughly disintegrated. Remove the crucible (and cover) wash inside and out with hot water, allowing the washings to run back in the beaker. Pour the solution into a good sized platinum dish, cover with a watch glass, and then add carefully, by raising the watch glass a little, hydrochloric acid in slight excess. Add a few drops of hydrochloric acid to the crucible and wash into the platinum dish. The cover should also be clean. After the effervescence is over, wash off the watch glass with hot water and place the dish in a hot air oven or on water bath and evaporate to dryness. It may be necessary to break the crust occasionally by stirring with a platinum rod which is afterward washed, the washings going into the dish. Dissolve the dried mass in water and again bring to dryness and heat in oven at about 110°C ., until all smell of hydrochloric acid is gone. Then add 10 c.c. strong hydrochloric acid and about 100 c.c. hot water. Warm. Filter off the insoluble silica, washing with hot water acidulated with a little hydrochloric acid, dry, ignite first with Bunsen then over the blast and weigh. Weight equals silica (SiO_2).

Oxide of Iron and Alumina.—Acidulate the filtrate from the SiO_2 with hydrochloric acid and concentrate to about 100 c.c. then add ammonia in slight excess. Boil until it smells

but slightly of it and allow to settle. Pour off the clear supernatant liquid, add about 100 c.c. of hot water, allow to settle and again decant. Repeat several times, stirring the precipitate and added water each time, then transfer precipitate to a filter using hot water and rubber tipped rod to clean the precipitate from the beaker. Wash precipitate once or twice with hot water, then drain well and ignite wet. Weight equals $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$. If it is desired to get the oxide of iron and alumina separately, mix the oxides with about 8 times their weight of carbonate of soda in a platinum crucible and fuse over the blast lamp. Digest crucible and contents in hot water, and when the fused mass is softened throw on filter and wash slightly with water, then dissolve in hydrochloric acid and estimate the iron in the solution either gravimetrically or volumetrically by permanganate or bichromate of potash.

The gravimetric method is often more convenient on account of its weighing the iron direct. In this method, after fusing the oxides, filtering, washing and dissolving in hydrochloric acid, the iron is separated from the alumina by precipitation with ammonium sulphide. Add to the solution containing the iron and aluminum as chlorides about five times the weight of the oxides, of citric acid, and excess of ammonia. The solution should remain perfectly clear. If it does not remain so add hydrochloric acid in slight excess, then a few grams more of citric acid and excess of ammonia. Now heat the clear solution to boiling and add solution of sulphide of ammonium in slight excess. Allow to settle and filter, washing the precipitate with water containing a little ammonium sulphide. Keep the funnel covered with a watch glass during the washing. When the precipitate and paper have been washed four or five times and the paper is drained quite dry, pour on the filter hot dilute hydrochloric acid, and allow the solution to run into the beaker in which the precipitate was made. Wash with hot water. Now add a few drops of nitric acid and evaporate to dryness in the beaker, dissolve in as little hydrochloric as is necessary to effect solution, dilute to about 25 c.c. and filter. Wash paper with hot water. Precipitate the iron with ammonia, boil, filter, wash, and ignite in the same manner as described for the estimation of total iron and alumina.

The weight equals ferric oxide (Fe_2O_3) which deducted from the total oxides of iron and alumina ($\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$) leaves the alumina (Al_2O_3).

Following is the method by using permanganate of potash. Prepare a solution of permanganate by dissolving 3.5 grams of the c.p. salt in one litre of water. When all is in solution pour it into a glass stoppered litre bottle and shake thoroughly. (After its equivalent in iron is found it should be labeled and kept in a dark place and its strength ascertained from time to time). Standardize the solution by means of ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$). Take exactly 5 grams of the c.p. salt and dissolve in 300 c.c. water, add 10 c.c. sulphuric acid and cool by allowing the hydrant water to run over the outside of the flask. Now fill a 50 c.c. burette (glass cock) with the permanganate solution exactly to the mark and allow it to run into the ferrous sulphate solution, drop by drop, until a faint pink tint is obtained, which shows that the iron is all oxidized and the one drop excess of permanganate colors the solution. It is best to repeat the operation several times and take an average.

Pure ferrous sulphate contains 20.1439 per cent. of iron (Fe) then .5 grams contains .1007 grams iron and if this took 15 c.c. of the permanganate solution then 1 c.c. would equal .0067 grams iron. With this solution we can now find the amount of iron in an unknown solution. To do this the iron must first be reduced to the ferrous condition by means of metallic zinc or some other deoxidizer. To use zinc transfer the solution of iron chloride obtained in the above fusion to a flask of about 500 c.c. capacity and add 3 grams (roughly weighed) of the granulated zinc, warm slightly, and allow the action to go on until the solution is perfectly colorless. Then add a few c.c. of hydrochloric acid to dissolve the remaining bits of zinc and if the solution still remains colorless it may be considered deoxidized. If not, more zinc must be added and the action allowed to continue. When the solution is completely deoxidized add a mixture of 10 c.c. sulphuric acid and 10 c.c. water, wash off the funnel allowing the water to run down the neck of flask and then cool by allowing the hydrant water to run on the outside of the flask. Now run in the standard solution of permanganate from the burette until the pink tint is ob-

tained. The amount of permanganate solution used, less correction for zinc, is then read and knowing its strength in iron the amount of iron in the sample is readily calculated. If we found it required 54 c.c. standard permanganate solution with a strength 1 c.c. = .0067 grams Fe., then $54 \times .0067 = .3618$ grams Fe., which multiplied by factor 1.428 gives .517 Fe_2O_3 . Having now the weight of oxide of iron and alumina and oxide of iron the difference equals alumina.

Calcium and Magnesium.—Concentrate the filtrate and decantation water from precipitation of the iron and aluminum first adding a little hydrochloric acid, to about 100 c.c. and proceed as detailed in the analysis of "Whiting" for the estimation of calcium and magnesium.

Potassium and Sodium.—The following method known as the J. Lawrence Smith method will be found probably as expeditious and accurate as any for the determination of the alkaline salts. Weigh out 1 gram of the clay and rub up in a mortar first with about a gram of c. p. ammonium chloride and then add 10 grams c. p. carbonate of calcium and mix thoroughly. Transfer to a large platinum crucible, cover and heat for a few moments to decompose the ammonium chloride. Now place the crucible on the triangle and adjust the flame so that the crucible is cherry red and keep at this temperature for about one hour. Remove flame and cool. Place crucible with contents and cover in a beaker, add about 100 c.c. of water and heat until the mass is thoroughly slaked. Take out the crucible and wash with hot water, allowing water to run back in beaker and filter. The filtrate contains the alkalies with impurities. Add about 2 grams c.p. ammonium carbonate and about 5 drops of ammonia and evaporate to about 25 c.c. Filter off the calcium carbonate, wash slightly with water. Add more ammonium carbonate to make sure that all the calcium has been precipitated. If a precipitate appears, evaporate to a small bulk and again filter. Usually two or three treatments will take out all the calcium. Now add a few drops of hydrochloric acid, evaporate to dryness in a weighed platinum dish, then heat carefully not quite to redness until white fumes of ammonium chloride cease to come off, cool and weigh as chlorides of sodium and potassium ($\text{NaCl} + \text{KCl}$). Add a few c.c. of water to the dish and the

chlorides should dissolve completely to a clear solution. If any insoluble matter appears, it must be filtered off and the filter washed once or twice with hot water and the filtrate again brought to dryness and weighed as before. If no insoluble matter appears add a solution of platinic chloride in excess and evaporate in a water bath until it begins to cake, then add about 20 to 50 c.c. of 80 per cent. alcohol to dissolve the excess of platinic chloride, stir and allow the crystals of potassium platinic chloride to settle, filter in a previously dried and weighed filter washing with 80 per cent. alcohol until the washings come through colorless. Dry in oven at $110^{\circ}\text{C}.$, and weigh quickly. Weight equals the potassium platinic chloride (K_2PtCl_6), multiply by factor .1939 gives K_2O . Then multiply K_2PtCl_6 by factor .3055 which will give KCl. Deduct this weight from the total KCl and NaCl, difference equals NaCl, which multiplied by the factor .5306 gives Na_2O .

Water.—To estimate the combined water ignite one gram of the clay to redness in a platinum crucible until the weight remains constant. The loss equals water.

The following analysis of China clays are given by Hurst:

	Cornwall.	China.	America.	French.
Silica (SiO_2).....	46.78	50.50	47.13	48.37
Alumina (Al_2O_3).....	39.60	33.76	36.76	34.95
Water (H_2O).....	13.16	11.22	15.13	12.62
Potash (K_2O).....	0.12	1.90		1.76
Ferric Oxide (Fe_2O_3)...	0.09	1.80	trace	
Lime (CaO).....	0.07		0.04	0.24
Magnesia (MgO).....		0.80		
	99.82	99.98	99.06	97.94

4. *Silica (Silix.)*—Silica is a natural product of the composition SiO_2 , has quite an extended use as a pigment and is used largely in the manufacture of wood fillers. It is light, white, inert and insoluble in acids. Some of the brands are made by pulverizing crystallized quartz (which is pure silica) and others are found as powder of sufficient fineness for use without any further preparation. As silica is found in a pure condition an analysis is not often required. Occasionally the relative purity of several brands may be desired in which case the determination of silica alone will be suffi-

cient. Other constituents may be lime and magnesia, iron and water, with possibly traces of alkalies. For the analysis proceed as detailed in the analysis of China clay, page 10.

ANALYSES OF SILICA BY THE AUTHOR.

	No. 1	No. 2	No. 3	No. 4
Silica	99.40	97.70	98.01	98.14
Moisture	.12	undet		.32
Potassium Oxide	undet	undet	.14	.24
	99.52	97.70	98.15	98.70

5. **Barium Sulphate** (*Barytes, Blanc Fixe.*)—Barium sulphate is used largely as a cheapener in paints but its analysis is not very often required. It consists of about 98 to 98.8 per cent. barium sulphate in the best grades, with small amounts of calcium sulphate, silica or clay, iron and alkaline salts. It is usually desired to ascertain only the percentage of barium sulphate (BaSO_4) for which the following plan of analysis may be used:

Barium Sulphate.—One gram of the dried sample is mixed in a glass or agate mortar with seven grams sodium carbonate and three grams potassium carbonate and then fused over a blast lamp in a platinum crucible, and the crucible and contents when cool, placed in a beaker and covered with water. Now digest until the fused mass is detached from the crucible and the barium carbonate that is formed has settled. Filter and rub the crucible clean with a rubber-tipped rod. Wash several times with hot water. Dissolve the barium carbonate on the filter with warm hydrochloric acid (1 part acid, 3 parts water) placing a watch crystal on top of funnel to prevent loss from effervescence. Repeat the acid treatment until all is dissolved, and thoroughly wash the paper with hot water. The solution now contains the barium as chloride. Heat to boiling, and while hot add dilute sulphuric acid (1 part acid, 3 parts water) with constant stirring until a precipitate ceases to form. Allow to stand about four hours in a warm place, then filter, washing the precipitate several times, first by decantation and then on filter with hot water. It is best to use a double filter as the precipitate has a great tendency to run through. Dry and ignite, first separating the precipitate as clean as possible from

the paper. Weight obtained multiplied by 100 gives the percentage of barium sulphate.

Qualitative Tests for Barytes.—To detect barium sulphate in a mixture of pigments, such as a mixed paint, dissolve about two grams in a mixture of 10 c. c. conc. c. p. hydrochloric acid and 30 c. c. water and boil for five minutes. Filter and wash the insoluble residue which may contain barytes, with hot water. Dry the precipitate, separate from paper and apply the following tests: Mix a small portion with dry c. p. sodium carbonate on a piece of stick charcoal (first digging out a round hole) moisten with water and fuse with a blow-pipe. When cool place the fused mass on a bright silver coin, add a few drops of water and if barium sulphate is present the coin will be blackened owing to the formation of silver sulphide. If lead sulphate or calcium sulphate were in the sample they will go into solution by the treatment with hydrochloric acid. The insoluble residue may be again confirmed by the flame test. Bend the end of a clear platinum wire into a small loop in which pick up some of the material to be tested and moisten with dilute hydrochloric acid. Now hold steadily in the outer edge of a Bunsen burner flame until a sharp point of the material is hot and if barytes is present a yellowish green tint will be imparted to the flame. This test is a little difficult to make at first as the tint appears in flashes, but with practice it will be found very decisive. It is a good plan to try the flame test, using pure barium sulphate.

In the presence of clay or silica the barytes may be in such a small amount that the flame test or blow-pipe test may not be decisive enough to pronounce the presence of barytes with certainty. In such cases it is best to fuse about one gram of the insoluble residue with sodium carbonate and proceed as detailed above for the estimation of barytes, and if it is desired to further confirm the precipitate obtained with sulphuric acid, it may be done by the flame or fusion tests.

6. *Magnesium Carbonate (Magnesite).*—*Insoluble Matter.*—Add about 20 c.c. water to one gram of the sample and then about 10 c.c. conc. hydrochloric acid, cover until effervescence ceases. Warm to complete the solution, allow to settle and filter, washing the filter thoroughly with warm water.

Dry, ignite paper and residue in platinum crucible. Weights equals insoluble matter.

Oxide of Iron and Alumina.—Add ammonia to the filtrate in slight excess, boil until the excess is driven off and filter, washing with hot water. Dry, ignite and weigh oxide of iron (Fe_2O_3) and aluminum (Al_2O_3). As the amount is usually small, their separation is not required.

Calcium Carbonate.—To the hot filtrate add ammonia to quite strong alkaline reaction, and if a precipitate formed, add ammonium chloride until dissolved. Now add solution of ammonium oxalate until the precipitate ceases to form and the ammonium oxalate is in excess. Let stand in warm place for four hours and filter, wash and ignite as in whiting. Multiply calcium sulphate (CaSO_4) by .7352 to convert to calcium carbonate (CaCO_3). If weighed as calcium oxide (CaO), use factor 1.7857.

Magnesium Carbonate.—Concentrate filtrate and washings to about 100 c.c., then add ammonia until the solution smells strongly of it and cool by standing beaker in a basin of cold water. When cold, add drop by drop with constant stirring a cold clear solution of sodium phosphate in excess and stir vigorously for several minutes, taking care that the stirring rod does not touch the sides of the beaker. Stand in a cool place over night. Filter off the supernatant clear solution and wash the precipitate several times by decantation, using a mixture of 1 part ammonia and 2 parts water.

Then transfer precipitate to filter, cleaning the sides of the beaker with a rubber tipped rod. Continue the washing on the paper until a few c.c. of the filtrate, to which has been added a drop of nitric acid, gives only a slight opalescence with a solution of silver nitrate. Now proceed to dry and ignite precipitate as directed in whiting, p. 9, calculating to magnesium carbonate.

Pure magnesite consists of 52.4 per cent. carbonic acid (CO_2) and 47.6 per cent. magnesia (MgO) and is rarely found in such purity, but contaminated with iron alumina, lime and moisture.

7. *Magnesium Silicate (Talc, Soapstone, Steatite.)*—This material is a natural product containing about 60% silica, 30% magnesium oxide and impurities, consisting of

small amounts of water, iron, alumina, calcium, etc. Its analysis is seldom required, but if desired the method given on p. 11 for china clay may be used, bearing in mind that the reagent, sodium phosphate, used in precipitating magnesium, must be largely increased to insure the complete precipitation.

To estimate the water, heat one gram in a porcelain crucible, using a strong heat of a blast lamp as the water is not expelled as readily as that in clay. Repeat the heating until the weight remains constant.

8. *Zinc Oxide (Zinc White).*—Zinc oxide is used to an enormous extent at the present day as a pigment, and is found in a majority of the mixed paints. Being an artificial product, its composition varies and the price is governed by the percentage of oxide of zinc it contains, the best grades approximating 100 per cent. zinc oxide, while some of the cheaper products contain more or less lead compounds, either as sulphates or oxides and possibly sulphate of zinc. The latter element is considered harmful, as it is soluble in water and is liable to cause the paint "streak." Zinc oxide is rarely adulterated.

Insoluble Matter.—For the analysis, take one gram of the previously dried (in air oven at 100° C.) sample and add about 100 c.c. hot water, then 10 c.c. strong hydrochloric acid and boil. The zinc oxide and sulphate of lead (also zinc sulphate and lead oxide) will go into solution and any insoluble matter such as sand or clay, as natural impurities, settle out. Filter on small ashless filter and ignite as in whiting, page 7.

Lead.—Add ammonia to the filtrate until nearly neutral, but is still acid, then pass a rapid stream of hydrogen sulphide gas into the solution until it is saturated. Warm and let stand until precipitate is settled. Filter and wash the precipitate with water containing hydrogen sulphide, allowing the wash water to run into the filtrate. The precipitate will contain the lead as sulphide. (See note 2.) Dry in air oven and ignite in a weighed porcelain crucible until the filter paper is charred. Cool. Add a few drops of conc. nitric acid to moisten, then add 5 c.c. fuming nitric acid and warm until sulphur is expelled, then add about 5 c.c. dilute sulphuric acid (1 part acid, 3 parts water) and heat gently until

nitric acid is driven off and white fumes of sulphuric acid appear. Now place on triangle and heat gently until the acid is entirely gone, then at a low red heat for a few moments, cool and weigh as sulphate of lead (PbSO_4 .)

Zinc Oxide.—Boil the filtrate from the lead precipitation to expel sulphuretted hydrogen, add a little chloride of ammonium, then ammonia to slight alkaline reaction, then colorless or slightly yellow ammonium sulphide in slight excess. Dilute to 250 or 300 c.c., cover and allow to stand in a warm place until settled. Filter, using a good sized filter paper and wash with water containing a little ammonium sulphide. (The slimy precipitate of zinc sulphide has a tendency to run through the paper. This may be greatly obviated by first pouring on the filter a solution of ammonium chloride. It is a good plan to change the beaker just before adding the precipitate so that should it run through, time will be saved from refiltering.) It is best to wash by decantation if the precipitate settles perfectly clear. When the precipitate is all on the filter paper and the wash water runs through, transfer the paper and contents to a small beaker and cover the precipitate with dilute hydrochloric acid, stand in a warm place until the smell of hydrogen sulphide is gone, then heat gently and filter, washing the filter with hot water. The zinc is then precipitated as carbonate. Add dry c.p. sodium carbonate from the end of a spatula (keeping beaker covered with a watch glass during effervescence) until the zinc is precipitated and the sodium carbonate is in excess. Add about 250 c.c. hot water and boil vigorously for about eight minutes, stirring occasionally to prevent bumping which is liable to occur before the boiling point is reached. The boiling renders the precipitate less flocculent and makes it settle much more readily. When the precipitate of zinc carbonate is *thoroughly* settled, pour off the *clear* supernatant liquid, and add to the precipitate about 200 c.c. more of hot water and stir vigorously. Allow to settle again and decant as before. After several decantings, pour the precipitate on a filter paper, and then rub the beaker clean with a rubber tipped rod and wash with water until the filtrate shows no alkaline reaction to test paper. The precipitate is then dried in the air oven and removed as free as possible from the paper

and placed on glazed paper. Ignite the paper in a weighed porcelain crucible and cool. Now add the precipitate and ignite gently at first and then at a red heat to constant weight. The carbonate is converted by the heat to the oxide (ZnO) and as such is weighed.

Sulphuric Acid.—One gram is dissolved in a mixture of 10 c.c. strong hydrochloric acid and 10 c.c. water and boiled until solution is complete. Filter off any insoluble matter, wash with hot water until all lead chloride crystals are dissolved. Keep solution hot, and add barium chloride until a precipitate ceases to form and stand in a warm place to settle. When completely settled, filter, keeping solution hot and wash with hot water until a drop of the filtrate fails to give a turbidity to a solution of silver nitrate. Ignite as in whiting and convert to sulphuric acid (SO_3) by multiplying by .3433.

Calculation.—Assuming that all the sulphuric acid will combine first with the lead, multiply the sulphuric acid (SO_3) found by 3.78 which gives lead sulphate. Deduct from total lead sulphate and if any lead sulphate remains, calculate to lead oxide (PbO) by multiplying by .7359. All the zinc then remains as zinc oxide in which form it is weighed. If, however, there is not enough lead to combine with all the sulphuric acid the remaining sulphuric acid is calculated to sulphate of zinc (ZnSO_4) by multiplying by 2.01. Calculate the sulphate of zinc to zinc oxide by multiplying by .503 and deduct from total zinc oxide. The difference is the zinc oxide in sample.

Note 1.—Care must be taken that lead sulphate is not mistaken for silica or clay, as it is not readily soluble in hydrochloric acid. Hard boiling will generally dissolve it. Should there be any doubt about the insoluble matter, pour on the filter a warm solution of ammonium acetate, wash well with hot water acidify the filtrate, then precipitate the lead with sulphuretted hydrogen, and proceed as detailed for the lead estimation.

Note 2.—If the precipitate is light colored it may be due to some zinc having precipitated or possibly free sulphur, due to the solution being too acid. In such cases the precipitate is filtered, washed from filter and boiled with a little dilute nitric acid, filtered, washed with hot water and the lead

again precipitated, after neutralizing the excess of acid with ammonia.

Note 3.—The sulphide of zinc, after drying, may be placed in a weighed porcelain crucible and ignited for some time at a red heat, allowing free access of air. The sulphide is “roasted” or converted to the oxide at once. Care must be taken to heat long enough to thoroughly convert it to the oxide, and several weighings should be made to insure a constant weight. This method is best when the zinc is present in small amounts.

Remarks.—In the majority of cases, it is generally desired to obtain the percentages of zinc (as zinc oxide) and total lead (as lead sulphate) not regarding the probable small amounts of lead oxide or zinc sulphate. In the best brands of zinc white, the two latter compounds need not be seriously considered, in fact, the problem of forming combinations of sulphuric acid, lead and zinc to give oxide and sulphate of zinc and oxide and sulphate of lead is a difficult one. But there are on the market pigments consisting of nearly equal parts of sulphate of lead and zinc oxide with several percentages of lead oxide and zinc sulphate, being the resulting product from roasting, volatilizing and condensing lead and zinc ores consisting largely of sulphurets of these metals.

As sulphate of zinc is soluble in water, a quick test might be proposed by leaching it out and then estimating the zinc in solution, but as zinc oxide is not absolutely insoluble in water a large excess must be avoided. Take 5 grams and brush into a filter and then fill the paper *twice* with warm water, using about 20 c.c. for each washing. Allow all water to drain and then precipitate the zinc in the filtrate with sulphuretted hydrogen after first making acid with acetic acid. Proceed now as in the estimation of zinc by “roasting” (note 3.) the sulphide to oxide and multiplying the zinc oxide by 1.987 to obtain the sulphate of zinc, and this result is multiplied by 100 and divided by 5 to get the percentage. In some instances, this method agrees very well with the long method detailed in the first part, but its usefulness can best be applied in a rough comparison when a number of samples are tested for “soluble zinc compounds.”

Rapid Method for Analysis of Zinc Oxide.—A method that is more rapid than the tedious precipitation of zinc as sulphide and quite accurate is the following:

Insoluble Matter.—Dissolve one gram in 7 c.c. conc. hydrochloric acid and about 10 c.c. water, boil for ten minutes. After any insoluble matter is settled, filter and wash filter with hot water. Dry, ignite and weigh insoluble matter.

Lead.—To the filtrate cautiously add about 3 c.c. conc. sulphuric acid, (or until lead ceases to precipitate) stir, evaporate to white sulphuric acid fumes. Cool. Add 10 to 20 c.c. water, stir to dissolve the zinc sulphate and allow to stand until the lead sulphate is completely settled. Then filter on Gooch crucible wash first with water containing 2% sulphuric acid then after removing the filtrate to a beaker rinsing out the flask several times with water, finish the washing with a mixture of half alcohol and half water. Dry, ignite and weigh lead sulphate.

Zinc Oxide.—To the filtrate in beaker containing the zinc sulphate add sodium carbonate in excess, boil, filter and wash with hot water, following the direction given on page 20 for estimating the zinc oxide.

Sulphuric Acid.—For the sulphuric acid proceed as directed on page 21. Having now the percentage of zinc, lead and sulphuric acid the calculations are made as indicated on page 21.

ANALYSIS OF ZINC PIGMENTS BY THE AUTHOR.

	Zinc White.	Zinc White	Zinc White	Zinc White
Zinc Oxide.....	82.84	96.00	93.23	93.23
Lead Sulphate.....	15.57	3.57	4.81	4.81
Zinc Sulphate.....	1.60			
Lead Oxide.....				1.96
	100.01	99.57	98.04	100.00
		"Leaded Zinc."	Zinc Lead	Zinc Lead
Zinc Oxide			54.51	52.10
Lead Sulphate		34.56	40.51	41.03
Zinc Sulphate			1.16	5.12
Lead Oxide				
			96.18	98.85

9. **White Lead (*Basic Carbonate of Lead.*)**—The actual composition of white lead varies somewhat from that of theory, and any material variance is supposed to affect its value as a pigment.

The theoretical composition is:

Lead Carbonate (PbCO_3).....	68.95 per cent.
Lead Hydroxide (PbH_2O_2).....	31.05 “ “
	100.00 “ “

or

Lead Monoxide (PbO).....	86.32 per cent.
Carbonic Acid (CO_2).....	11.36 “ “
Water (H_2O).....	2.32 “ “
	100.00 “ “

having, therefore, the formula $2\text{PbCO}_3\text{PbH}_2\text{O}_2$. The function of the lead carbonate seems to be to impart body and color, while that of the hydrate of lead is to form a chemical combination with the oil to greatly increase the covering properties.

In the analysis of white lead the carbonic acid is the usual constituent sought, and its determination is one requiring care and exactness of apparatus. The usual method is to calculate the carbonic acid to lead carbonate, subtract from 100, and express the difference as lead hydrate. The reason for this is that the estimation of the combined water requires heat to drive it off and the liability of the sample to contain carbonaceous compounds such as lead acetate, traces of oil, etc. would give erroneous results.

In some laboratories, however, the combustion method is used, as it gives quick and approximate results of sufficient accuracy to check purchases and guard against adulterating and for this reason the method is detailed on page 28.

If only the lead is desired, it may be obtained as follows:

Dissolve 1 gram in 5 c.c. nitric acid (1.42 sp. gr.) and 20 c.c. water and heat to solution. If any insoluble matter appears, filter and wash with hot water. Now add dilute sulphuric acid to filtrate, a few drops at a time, with constant stirring, until the white precipitate of lead sulphate ceases to form, and evaporate in hot plate to sulphuric acid fumes. Cool. Add cautiously 15 c.c. water and an equal bulk of alcohol, stir

thoroughly and allow to stand about an hour, or until supernatant liquid is perfectly clear. Filter in a weighed Gooch crucible. Wash with a mixture of one half water and one half alcohol. After four or five washings, suck dry, remove crucible, attach cap, and place on a hot plate for a few moments to dry, then ignite on triangle at a red heat for five minutes. Cool and weigh. Multiply weight of lead sulphate by factor .7359 which converts it to (PbO) lead oxide.

If for any reason the Gooch crucible is not at hand or inadmissible, and the lead sulphate must be filtered on paper, proceed as detailed above and wash the paper, after adding the precipitate, with the mixture of alcohol and water. Then dry in oven, remove the lead sulphate from the paper as clean as possible, placing in a large water glass, and ignite paper in a weighed porcelain crucible. When all the carbon is burnt off, cool the crucible, then add 3 or 4 c.c. water and about two drops of nitric acid (sp. gr. 1.42), and warm until all the metallic lead is in solution. Cool and add a few c.c. dilute sulphuric acid to precipitate the dissolved lead then evaporate on water bath until no more water or nitric acid fumes are given off. This may be done more rapidly by placing in the *warm* part of a hot plate but care must be observed that none of the contents spirts out. Now place crucible in triangle and heat by playing the flame gently, and when all the sulphuric acid is expelled heat at a red heat for a few minutes, and cool. Add the bulk of the precipitate and again heat to redness for about 5 minutes. Cool and weigh.

F. C. A. Meisel (School of Mines Quar. 1.04, Vol. XIV.) has given the result of an investigation for the estimation of water and carbonic acid in white leads by four methods: viz., Loss in ignition, loss by use of alkalimeter, measuring carbonic acid gas by volume, and absorption of carbonic acid in caustic potash solution, reaching the conclusion that the last method gives the most trustworthy results, although it possibly requires longer time.

Estimation of Carbonic Acid.—The carbonic acid is absorbed in the Liebig potash bulb A, which carries the calcium chloride tube to prevent any moisture from being absorbed from the aspirator. Connecting with the Liebig bulbs are two U tubes, D, D, in which is freshly made anhydrous sulphate of copper and pumice for the absorption of any traces of hydrochloric acid gas and moisture. H is a

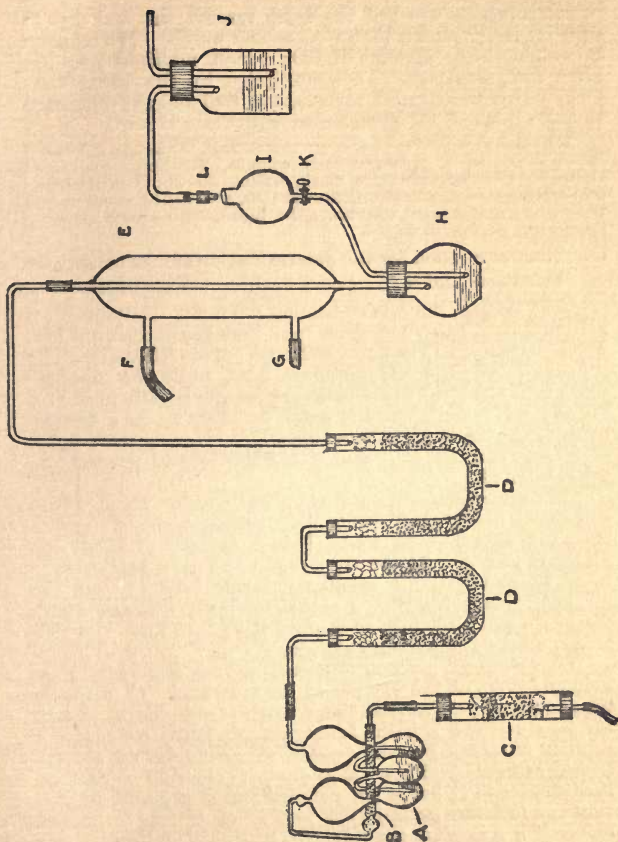
boiling flask with a capacity of 150 c.c., fitted with a rubber stopper with two holes in one of which passes the end of a glass condenser and through the other passes the bent glass tube of the bulb L. The latter has a glass stop cock at K. J is a wash bottle containing solution of caustic potash. The connection at L is made with a rubber stopper which fits tightly into the bulb I. M. is a rubber tube connecting the aspirator.

The caustic potash solution should have a specific gravity of about 1.27. This may be made by dissolving 30 grams of the stick potash in 100 c.c. water, which will make a solution of the desired strength.

The calcium chloride should be dried thoroughly at 200 c.c. (not fused); the white porous anhydrous chloride thus produced being very deliquescent it should be kept in glass stoppered bottle. The copper sulphate and pumice are prepared by heating pieces of each, which should be about the size of a pea, in a porcelain dish until the blue crystals are white, then transferred to a glass-stoppered bottle for keeping.

For the estimation of carbonic acid gas (CO_2), one gram of the sample is carefully brushed into H and after the Liebig bulbs are weighed the apparatus is connected, the water started in condenser E and the glass stop cock K in bulb I closed. Pour in bulb tube I diluted hydrochloric acid made up of 15 c.c. hydrochloric acid (1.2 sp. gr.) and 40 c.c. water. Insert the rubber stopper L securely in bulb I and open the stop cock K so that the acid will flow slowly in H. When nearly all the acid has passed into flask H close stop cock and apply a slow heat increasing gradually until the solution boils which will take about 10 minutes. A few moments further boiling will bring all the lead chloride in solution. When this is accomplished the flame is lowered and suction commenced. After 10 minutes the flame is withdrawn and the suction continued at the rate of about three bubbles per second for about 20 minutes, at which time the apparatus should be free from CO_2 .

Disconnect the potash bulbs and place in the balance case for about 30 minutes to get the same temperature as that of the case. At the end of this time if the levels of the



potash solutions are not the same remove the end caps to equalize the pressure, then replace and weigh. The increase in weight is the amount of CO_2 , which, multiplied by 100, gives the percentage. This, multiplied by the factor 6.068, gives the percentage of carbonate of lead and the difference between this and 100 is expressed as hydrate of lead.

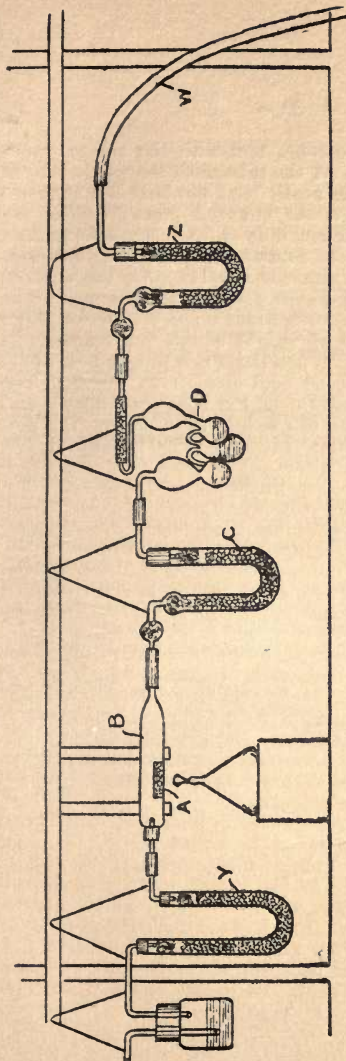
With due regard to the necessary precautions, the foregoing method for the estimation of carbonic acid is accurate. The apparatus should always be tested by running a blank, boiling the dilute hydrochloric acid solution (but of course omitting the sample) for 10 minutes, and aspirating about twenty minutes. The Liebig bulb should show no appreciable increase in weight.

Combustion method for the determination of lead oxide, carbonic acid and water in dry basic carbonate of lead.

The method consists of heating the sample contained in a platinum boat, in a short glass tube thus decomposing it into water, carbonic acid gas and litharge (oxide of lead). The litharge remains in the platinum boat, while the carbonic acid gas is absorbed in a solution of caustic potash in a Liebig absorption bulb, and the water is taken up in a U tube containing granulated calcium chloride. Having the weights of litharge carbonic acid and water the several percentages are calculated.

The apparatus is supported by the wooden stand. B is a piece of heavy glass combustion tubing about six inches long and sufficient diameter ($\frac{5}{8}$ inch inside) to admit the platinum boat A. At the right hand end is a rubber tube which fits snugly to the calcium chloride tube C. On the left hand side is inserted a rubber stopper, through which passes a small glass tube to connect the guard tube Y which is also filled with calcium chloride.

The platinum boat A is a small narrow box which must hold one gram of the sample to be examined. C is the U tube containing dried granulated calcium chloride to absorb the moisture, and D is the Liebig potash bulbs containing a solution of caustic potash in water, sp. gr. 1.2 for the absorption of carbonic acid gas. Z is another chloride of calcium tube similar to C, but is not weighed and is a guard to prevent any moisture getting back into D or C. As explained before Y is a calcium chloride tube to dry the atmospheric air



before entering the combustion tube, and X is a small bottle containing a strong solution of potash to free the air from carbonic acid. W is a rubber tube connecting the aspirator bottle to draw air through the apparatus. All chloride of calcium tubes should have a plug of cotton at the ends to prevent any of the chloride from being carried out.

Detach C and D and wipe carefully with dry cloth. Both ends of each should be carefully closed by small pieces of solid glass tubes inserted in the pieces of rubber tubing. Next weigh. Then weigh the platinum boat and in it weigh one gram of the sample. Detach combustion tube and gently heat over a naked flame until the tube is hot, then suck the air through by the mouth — this being necessary to remove any moisture. Now connect the whole apparatus in the position as seen in the cut. Draw air through by means of the aspirator bottles at the rate of a few bubbles per second. Note whether there are

any leaks, and if there are none, heat with the alcohol lamp slowly along the bottom of the tube until the whole tube is hot. Place the lamp underneath boat and heat for about 20 minutes or until the lead has changed from white to the red color of litharge. When it is of uniform color remove the lamp and allow air to aspirate for about 10 minutes. Disconnect the calcium chloride tube C, plug the ends as before the operation, and do the same with the potash bulbs D and weigh. Observe precautions on p. 26, for weighing the bulbs. The increase of weight equals the amounts of carbonic acid and water. When these are weighed the boat containing litharge will be cool enough to weigh. The difference between the weight of the boat plus litharge and the weight of boat equals the weight of litharge. We now have the following weights: Litharge (PbO), water (H_2O), carbonic acid gas (CO_2).

Qualitative Tests of White or Tinted Mixed Paints, Paste Paints, Fillers, Etc.

The readiness of lead sulphate and calcium carbonate to react, when the latter is in excess, in treatment with dilute acetic acid precludes the use of acetic acid as a general solvent for mixtures of pigments.

When the above mentioned pigments are so treated, lead carbonate and calcium sulphate are formed and both go into solution and part if not all of the lead existing as sulphate will be figured with the soluble lead pigments such as lead oxide or carbonate.

Acetic acid may be used, however, when either one of them only is present and although lead sulphate is slightly soluble the result will be accurate enough (especially enough when sulphuric acid is also estimated) for the purposes for which an analysis of a mixtures of pigments is used.

As there are on the market, and in large use, pigments that contain considerable lead sulphate such as "sublimed lead," "leaded zinc," "zinc lead," "zinc oxide," it is obvious that acetic acid as a solvent cannot be used to advantage except in the mixtures in which lead sulphate and calcium pigments are present either singly or not at all.

For this reason it will be necessary to vary the method employed in accordance with preliminary tests.

Of course the regular methods of analysis may be used in a mixture of pigments by estimating the acid radicals and bases, but the time involved is much longer and the tedious separation of lead and zinc by the use of sulphuretted hydrogen and ammonium sulphide in mineral acid solution is avoided. Then, too, the combinations to arrive at the different pigments used is often a matter of conjecture. In the foregoing the combinations are much more rapidly obtained particularly in scheme I.

CHAPTER III.

WHITE AND TINTED PAINTS.

Qualitative Analysis of White or Tinted Paints, Paste Paints, Fillers and Enamel Paints.

Boil about one gram of the extracted sample with from 25 c.c. to 50 c.c. of acid ammonium acetate (see note 1) for 15 minutes. Filter and wash with hot water. Note whether effervescence takes place, indicating carbonates.

RESIDUE.

Ignite residue in a porcelain crucible. When cool test portion by flame test for barytes. If silica or China clay is suspected mix residue with five or six times its weight of sodium carbonate in a platinum crucible. Fuse. When cool place crucible and contents in beaker and cover the crucible with water and boil until the mass is disintegrated. Filter. Dissolve the barium carbonate on filter in few c.c. hydrochloric acid and allow to run in another beaker. Then add sulphuric acid in slight excess which will throw down any barium as *barium sulphate*. See Note 1.

To the filtrate from the barium carbonate and barium sulphate add hydrochloric acid in slight excess, place in a platinum dish and boil rapidly to dryness on a hot plate or directly over a low Bunsen burner flame. Heat until hydrochloric and sulphuric acids are gone and cool. Moisten with hydrochloric acid, add about 25 c.c. water and warm to dissolve. Any insoluble residue is *silica*. Filter, and to filtrate add ammonia in slight excess and boil. A precipitate of aluminum hydrate together with the presence of *silica* indicates *China clay*. Now add ammonium oxalate, stand a short time and, if calcium oxalate is precipitated, filter. Cool filtrate add ammonia and sodium phosphate, and if a precipitate of ammonium-magnesium-phosphate appears after standing a few hours it indicates presence of *magnesium silicate*. See Note 2.

FILTRATE.

Divide the filtrate in three nearly equal parts and proceed in each as follows:

Test for sulphates by adding a few drops of hydrochloric acid, heat to boiling, then add a few c.c. of barium chloride. A precipitate of barium sulphate indicates presence of *lead sulphate* or possibly *calcium sulphate*.

See Note 3.

Add a few c.c. conc. sulphuric acid or until the addition of a few drops ceases to form a precipitate of *lead sulphate*. Filter. To the filtrate add ammonia in excess, then a few c.c. ammonium sulphide. A white precipitate indicates presence of *zinc oxide*.

See Note 4.

Ammonia is added in excess, then ammonium sulphide until a precipitate ceases to form. Warm and stir to collect precipitate. Filter. To the warm filtrate add ammonium oxalate in excess, heat to boiling point and filter off calcium oxalate indicating presence of *calcium carbonate* or *calcium sulphate*. Cool the filtrate and add solution of sodium phosphate, stir and stand in a cool place for about thirty minutes and if any magnesium is present a precipitate will form indicating *magnesium carbonate*.

See Note 5.

Note 1.—Acid ammonium acetate solution may be prepared by mixing 15 parts water with 30 parts ammonia (sp. gr. 0.90) to which is added 50 parts glacial acetic acid. Test the solution with barium chloride, which should give no trace of a precipitate. About 70 c.c. of this solution will dissolve one gram of lead sulphate in boiling ten to fifteen minutes.

Note 2.—If any silica, other than that in the clay, is suspected it will be necessary to make a quantitative analysis in order to determine its percentage.

Note 3.—Calcium sulphate (gypsum) is used in paints to some extent and its presence complicates an analysis when whiting or lead compounds are present. It cannot be detected by dissolving the gypsum in water as might at first be supposed, owing to the reaction that takes place between sulphates and carbonates of lead and calcium. The fact of this peculiar reaction is brought out by G. W. Thompson in

the "Journal of the Society of Chemical Industry, Nov. 30, 1896," and the method here used for determining mixtures of sulphates and carbonates of lead and lime pigments by nitric acid and alcohol is based on the method given there. To test for the presence of gypsum proceed as follows: To about 1 gram add 20 c.c. of a mixture of 9 parts alcohol (95%) and 1 part nitric acid (1.42 sp. gr.) and after stirring allow to stand about 20 minutes. Then decant on a filter and again treat with the nitric acid-alcohol mixture, allowing to stand and decant as before. Repeat the treatment four times then pour any residue on filter and wash residue with nitric acid-alcohol mixture. If gypsum was present it will remain undissolved, while only the calcium carbonate (with possibly some of the zinc and lead salts) will go into solution. Now wash the residue of gypsum and other insoluble matter into a beaker with the aid of a wash bottle, add a few c.c. hydrochloric acid, boil and filter. To the filtrate add ammonia in slight excess, then ammonium sulphide, and warm until precipitate is settled. Filter. To the warm filtrate add ammonium oxalate and warm a few moments. A white precipitate of calcium oxalate indicates the presence of calcium which existed as sulphate.

Note 4.—If lead is present it may be as the basic carbonate, lead sulphate or lead oxide. Only a complete quantitative analysis will distinguish in which form the lead may be.

Note 5.—If only traces of magnesium carbonate are found they are probably due to impurities in the whiting. It is sometimes used in woodfillers.

Note 6.—Zinc sulphite and lead sulphite are seldom if ever found in American paints and for this reason have not been considered herein.

Having now the qualitative tests the scheme to use is determined by the following conditions:

USE SCHEME I.

If sulphates are present but no calcium pigments.
(Indicating sulphate of lead and absence of calcium carbonate or calcium sulphate.)

Or, if calcium pigments are present but no sulphates.
(Indicating calcium carbonate and absence of calcium sulphate and lead sulphate.)

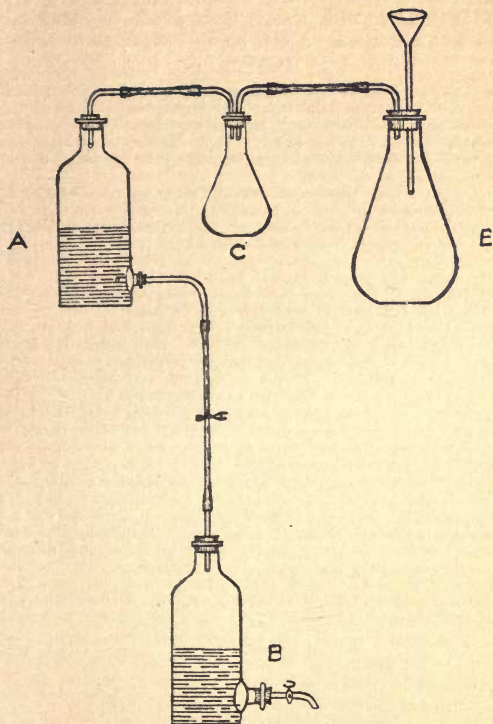
USE SCHEME II.

When sulphates and calcium pigments are present.

NOTE.—The above does not include the insoluble pigment, barytes.

A Gooch crucible is quite indispensable for the rapid filtering and ignition of precipitates, and is particularly useful in igniting precipitates that are liable to be reduced by filter paper, such as lead sulphate. The asbestos of the silky, fibrous kind should be previously digested for several hours in dilute hydrochloric acid, then washed by decantation with distilled water until free from acid, and thrown on a filter to drain and dry, then kept in a wide mouth bottle. To prepare the asbestos pad in the Gooch crucible, mix several grams (or a good sized "pinch") in a small beaker with distilled water and pour in crucible applying gentle suction. When quite dry by means of the finger press down the edge of the asbestos so that the whole pad has an even surface and is about an eighth of an inch in thickness. Now apply pressure until quite free from water, remove from funnel, attach the platinum cap and place on hot plate to dry, then place on triangle and ignite for about five minutes over flame at red heat. Cool, remove cap and weigh.

If for any reason the Gooch crucible is not admissible and it is desired to use a paper filter in preference to the asbestos pad, a perforated platinum cone may be used to prevent the paper being broken by the pressure of the pump. A funnel should be selected that the cone exactly fits so that the paper will not be broken from the sharp edge. Fold the paper and place in funnel, then fill the funnel with water and apply the suction. The paper will then be drawn tightly to the cone and funnel and is ready for use. The use of filter pumps to facilitate filtering operations has now become quite general and in most laboratories are indispensable. The kind of pump is generally determined by the water supply. The Richards injector is generally used where the water supply is sufficient. A very serviceable and simple pump may be arranged by using aspirator bottles as shown in cut on p. 37. The aspirator bottles A and B connect by means of a rubber tube with C which is an empty guard flask. It is necessary that the fall of water between A and B should be a considerable distance, about seven feet being generally sufficient. On the tube connecting A and B is a clamp for regulating the flow. The guard flask C is connected by another rubber tube with flask E. In the neck of E is a funnel for holding either the platinum cone or crucible. The water flowing from A to B causes a partial vacuum and creates a pressure on the filter. As soon as the water has run from A to B they are quickly reversed and the flow resumed, the same water being used over again. It is obvious that for filtering corrosive liquids such as nitric acid, this method cannot be used to advantage, but for lead sulphate in sulphuric acid or zinc carbonate, etc., it will be found very serviceable.



2. SCHEME I.

Boil one gram of the sample in 5 c.c. glacial acetic acid and about 25 c.c. of water for 10 minutes. Wash down the sides of the beaker with water, then dilute to about 50 c.c. and stand until settled. Filter on a 9 c.m. filter.

RESIDUE.

The residue insoluble in acetic acid may consist of either lead sulphate, china clay, barium sulphate, silica or magnesium silicate.

In order to effect a decomposition and solution it will be necessary to fuse in a platinum crucible which is impracticable when lead sulphate is present. To remove the lead sulphate either one of two methods may be used:

1.—By converting to lead carbonate and dissolving in dilute acid p. 39.

2.—Dissolving at once in hot ammonium acetate p. 39.

In either case the insoluble clay, silica, barytes, magnesium silicate will remain to be treated as directed on p. 40.

FILTRATE.

Lead acetate $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$, Zinc acetate $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2$, Calcium acetate $\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2$, Magnesium acetate $\text{Mg}(\text{C}_2\text{H}_3\text{O}_2)_2$.

Pass a rapid stream of sulphuretted hydrogen gas until saturated, warm and allow the precipitates of lead and zinc sulphides to settle quite well. Filter on a double filter using a platinum cone and pump, and when the supernatant liquid is filtered add about 20 c.c. sulphuretted hydrogen water. Stir and again allow the bulk of the precipitate to settle. Filter and repeat the washing by decantation four times. Now run the precipitate on the paper and by the aid of the wash bottle containing sulphuretted hydrogen spirit any loose precipitate on the filter disregarding what adheres to the beaker. Wash once or twice on filter with sulphuretted hydrogen water and suck quite dry.

PRECIPITATE.

Lead sulphide (PbS), Zinc sulphide (ZnS), p. 41.

FILTRATE.

Calcium acetate, $\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2$,
Magnesium acetate, $\text{Mg}(\text{C}_2\text{H}_3\text{O}_2)_2$,
p. 42.

REMOVING LEAD SULPHATE FROM RESIDUE INSOLUBLE IN ACETIC ACID.

Method I.—By Ammonium Carbonate: Plug up the end of the funnel with a filter paper tightly rolled or a better plan is to slip on the end a piece of rubber tubing about an inch long and close up the other end of the rubber tube with a bit of solid glass rod. Now place on the filter two or three grams of ammonium carbonate and fill the filter with warm water within a half inch of the top. Cover with a watch glass and allow to stand in a warm place for six to ten hours—best over night. The lead sulphate is by this means converted to lead carbonate. Now pull off the rubber plug and wash the filter with warm water. Reject filtrate. Dissolve the lead carbonate by pouring warm acetic acid on filter quickly covering with watch glass to prevent loss by effervescence. Wash off watch crystal, allowing the water to run on the filter and then wash out the lead acetate with hot water. It is a good plan to add acetic acid a second time to make sure that all the lead carbonate is dissolved. Proceed with the residue as described on p. 40.

Method II.—By Ammonium Acetate: Remove filter and contents from funnel, place in a beaker and add from 30 c.c. to 50 c.c. acid ammonium acetate solution, (see note I.) and boil for 10 minutes. Then filter any insoluble residue (containing first filter paper) and wash thoroughly with hot water. The lead will now be in the filtrate. The residue is treated as described on p. 40.

Determination of Lead in Solution obtained by either Method I or II.—Pass a stream of sulphuretted hydrogen gas through the solution until saturated and filter. Reserve filtrate. If there is considerable lead sulphide proceed exactly as described on p. 24 for the estimation of lead as sulphate, using a Gooch crucible. The lead is weighed as sulphate (PbSO_4) and exists as such in the sample. Should the precipitate of lead sulphide be small it may be converted directly to the sulphate as follows: Dry precipitate in an air oven and carefully transfer from the paper to a piece of glazed paper or watch glass. Now fold the paper and place in a weighed porcelain crucible and ignite until charred. Add the precipitate and moisten all with nitric acid (1.42 sp. gr.) Heat gently until the carbon is consumed, cool and add about 5 c.c. fuming nitric acid, cover with small watch crystal and

heat until the free sulphur is oxidized. Then add a drop or two of sulphuric acid and heat as usual, finally cooling and weighing the lead sulphate.

Filtrate.—The filtrate from the lead sulphide precipitate is boiled to expel sulphuretted hydrogen, then ammonia added in slight excess and ammonium oxalate. If a precipitate appears it is probably due to calcium carbonate in the china clay or possibly whiting that failed to dissolve in the acetic acid. If the sample contains whiting, that found in the insoluble residue is added to it but if no appreciable amount is present it is added to the china clay or if desired it reported singly. China clay or silica of good quality does not contain calcium soluble in acetic acid.

Residue Insoluble in Acetic Acid After Removing Lead Sulphate.—Dry the residue left after removing the lead sulphate, clean precipitate as free as possible from the paper, ignite paper in a weighed porcelain crucible, cool, add precipitate and heat to redness for ten minutes. Weight equals barytes, silica or china clay. Transfer to a capacious platinum crucible, add ten times its weight of sodium carbonate, fuse and proceed as directed in china clay p. 11. After the mass is thoroughly disintegrated, filter off the insoluble barium carbonate, wash with hot water (reserving filtrate) and dissolve on filter with dilute hydrochloric acid. Finally precipitate with sulphuric acid as directed on p. 16, which is the barium sulphate existing as such in the sample.

If the barium sulphate be deducted from the total insoluble matter the difference is the china clay and silica. If, however, a more accurate result is desired unite the filtrates from the barium carbonate and barium sulphate precipitations transfer to a platinum dish evaporate to dryness first on a water bath and finishing on a hot plate. Heat until no more sulphuric acid fumes are given off. Cool. Add sufficient water and heat in water bath until all is in solution except the silica, then add a drop or two of sulphuric acid and again bring to dryness and heat until all acid fumes are driven off. Now add water and proceed as directed in china clay p. 11. for the estimation of silica and alumina. The average composition of china clay is 2 SiO_2 , Al_2O_3 , $2 \text{ H}_2\text{O}$ containing 39.53 per cent. of Al_2O_3 . Multiply the Al_2O_3 found by 2.537 and

the result will be china clay. Then multiply the china clay found by factor .465 and the result will be the silica in the china clay and if the difference exceeds 5 per cent. it may be due to free or added silica.

If the qualitative examination shows magnesium present in the insoluble residue then it will be necessary to proceed with the filtrate after filtering off the aluminum. This is done exactly as detailed in china clays for the estimation of calcium and magnesium. When such complex insoluble residues are found and this is rarely the case, it is a difficult problem to form an opinion or give percentage of the original products. In such cases it is the better plan to give the percentage found such as silica, aluminum oxide, magnesium oxide, barytes, etc., with possibly an attempt to combine the percentages to give probable products, which may be free silica, barytes, china clay, magnesium silicate, etc.

Precipitate of Lead Sulphide and Zinc Sulphide.—

Remove the filter and contents from the funnel and spread out on a glass plate or the concave side of a large watch glass. Now with a wash bottle containing hot water spirit the precipitate from the paper into the beaker in which the precipitation was made. Add moderately strong hot nitric acid to the paper which dissolves the remaining stains of the sulphide and allow to run in beaker. Finally, wash the paper with hot water until it has no acid reaction. (The paper is now rejected.) Boil until all is in solution and only the light colored sulphur remains. If the precipitate is dark colored, due to undissolved lead sulphide, add a few c.c. of conc. nitric acid and again boil which will usually dissolve the last traces of the sulphides. Now filter and wash the sulphur until free from acid and if filtrate exceeds 50 c.c., boil to about that volume and cool.

Precipitate lead by adding cautiously 5 c.c. conc. sulphuric acid and evaporate as rapidly as possible without bumping. When the dense white fumes of sulphuric acid appear, allow to cool. Add 25 c.c. water, stir to dissolve all zinc sulphate, then stand until perfectly clear and the lead sulphate is settled. Filter in a Gooch crucible, wash with 2% solution of sulphuric acid and water, until all sulphate of zinc is washed out, then remove filtrate and finish the washing with a mixture of half alcohol and half water, proceeding as directed on p. 24 for the estimation of lead. Multiply the

lead sulphate by .852 to convert to $2\text{PbCO}_3\text{PbH}_2\text{O}_2$. (*Basic carbonate of lead.*)

Dilute filtrate to about 400 c.c., heat nearly to boiling, then proceed with the estimation of zinc by precipitating with sodium carbonate, finally igniting and weighing as *zinc oxide* (Zn O) which exists as such in the sample. Follow directions for estimation of zinc on p. 20.

Filtrate Containing Calcium Acetate and Magnesium Acetate.—Transfer the filtrate to a beaker and rinse out the flask which contained it several times with water, adding the washings to the beaker, and boil down to about 75 c.c. If sulphuretted hydrogen is not entirely disappeared, continue boiling until but a very faint odor is perceptible. Now add ammonia in excess and to the hot solution add, drop by drop, with constant stirring, solution of ammonium oxalate, and allow to stand on the edge of the hot plate for about four hours. Filter the calcium oxalate, washing with hot water, dry, ignite and weigh as calcium sulphate, calculating to *calcium carbonate* (CaCO_3) as directed on p. 8.

If magnesium is present, evaporate the filtrate to about 50 c.c., cool and proceed as directed on p. 9, for the estimation of magnesium. Weight of magnesium pyrophosphate ($\text{Mg}_2\text{P}_2\text{O}_7$) obtained is calculated to *magnesium carbonate* (MgCO_3) by multiplying by .7567.

Note.—If barium carbonate is present it will be necessary to first separate it from the calcium and magnesium by adding dilute sulphuric acid drop by drop until a precipitate ceases to form, allow to stand in a warm place until the precipitate is pretty well settled, then filter and proceed as in barytes for the washing ignition and weighing of the barium sulphate (BaSO_4). Multiply by .845 to convert to barium carbonate (BaCO_3). It is essential that the solution be kept quite dilute and a few c c. hydrochloric acid added to prevent any calcium if present from precipitating as sulphate.

Notes on Scheme I.

Note I.—Owing to the probable presence of “sublimed lead” or products containing lead oxide, it will be necessary, where it is suspected, to make an estimation of the carbonic acid present, which may be done by the apparatus and method described on p. 25. Then having the carbonic acid (CO_2) calculate it to basic lead carbonate by multiplying by 8.8. Convert the basic carbonate of lead found to lead

sulphate and deduct from the total lead sulphate (acetic acid solution), and if there is any excess of lead sulphate (say one per cent.) it is calculated to lead oxide (PbO). Should whiting be present it will be necessary to first satisfy it with carbonic acid by multiplying the calcium sulphate (CaSO_4) found by .735 (of if CaO by 1.885) which equals whiting (CaCO_3). Now find the carbonic acid (CO_2) in the whiting by multiplying by .44, and deduct from the total carbonic acid found. If magnesium carbonate is present, calculate its carbonic acid by multiplying by .52 and deduct from the CO_2 left. Then calculate the balance of the CO_2 to basic carbonate of lead by factor 8.8. Any lead left unsatisfied is calculated to lead oxide (PbO) by deducting the lead in the white lead from the total lead found soluble in acetic acid.

Note II.—If the sample be a tinted paint, the coloring or tinting pigment is usually found by difference, that is, the total result by analysis is subtracted from 100. In tinted or mixed paints it does not exceed but a few per cent. and the pigment used in coloring is disregarded.

Note III.—If the sample consists of only white lead and zinc oxide, dissolve in acetic acid as usual, filter, and instead of precipitating as sulphides the lead may be separated at once by adding sulphuric acid and evaporating as usual, and then precipitating the zinc in the filtrate. The lead sulphate if present will remain insoluble in the filter and is treated in the usual way. As lead sulphate is not absolutely insoluble in acetic acid particularly when it is present in large amounts, it is well to make a determination of the sulphuric acid and calculate to (PbSO_4) lead sulphate, then deduct from the total lead sulphate (both soluble and insoluble in acetic acid) and the balance is calculated to white lead.

Note IV.—In the analysis of complicated mixtures of pigments the chemist has to assume some arbitrary formulae such as that of white lead which do not always conform to the theoretical formula, but are liable to vary somewhat. So, too, in the estimation of china clay. Yet there is no ready method that will give these in their exact composition and neither is it necessary in the analysis of a paint for practical purposes.

3. SCHEME II.

For mixtures containing sulphates or carbonates of lead and calcium together with other pigments.

Carbonic Acid.—If carbonic acid is present it is estimated by the method and apparatus as described on p. 25.

Sulphuric Acid.—Determine as detailed on p. 21, weighing as barium sulphate, convert to SO_3 by factor .3433.

Bases.—Boil one gram 20 minutes with about 40 c.c. acid ammonium acetate solution, keeping the beaker covered. Filter and wash filter thoroughly with hot water.

INSOLUBLE.

Barytes, china clay, silica and magnesium silicate. Dry, ignite and weigh, then proceed as detailed for the separation and estimation of silica, aluminum, barium, calcium and magnesium. p. 40.

FILTRATE.

Lead, zinc, calcium and magnesium acetates. Proceed as detailed for their separation and estimation. Pages 41 and 42.

Notes on Scheme II.

Note I.—If the qualitative test shows that lime exists both as the carbonate and sulphate together with lead sulphate (and the latter is generally present in mixed paints), it will be necessary to extract and estimate the lime carbonate, and having the total lime, that existing as sulphate may be calculated. The following method due to Thompson is probably the best.

Proceed as follows: One gram of the sample is treated in a small beaker with 25 c.c. of a mixture of 1 part nitric acid (1.4) and 9 parts 95% alcohol, and allowed to stand without heating for 20 minutes. Then decant the clear liquid on a dry filter and again treat with the alcohol-nitric acid mixture, allow to stand and filter as before, repeating the operation four times. Finally wash the paper with the mixture. The calcium carbonate together with some lead and zinc will go into solution, while any gypsum and insoluble matter will remain on filter and is disregarded. Now evaporate the filtrate to dryness to expel alcohol, and add sulphuric acid and heat to fumes. Cool. Add a few c.c. nitric acid, then ammonia to alkaline reaction, then acetic acid in excess, and boil to solution. While hot pass sulphuretted hydrogen gas until

saturated and filter off any lead or zinc. Wash precipitate, first by decantation, then on filter with water containing hydrogen sulphide, and evaporate filtrate and washings until sulphuretted hydrogen has disappeared. Make alkaline with ammonia if not already so, precipitate the calcium with ammonium oxalate, allow to stand several hours in a warm place, filter, wash, ignite and weigh as calcium sulphate.

Note II.—As basic carbonate of lead does not conform strictly to the theoretical formula any excess of lead after satisfying the carbonic acid and sulphuric acid does not necessarily imply the presence of lead oxide. The latter is usually present in sublimed products or in the presence of much lead sulphate. It is possible that a very small percentage of lead in the drier may not have been completely washed out with the gasoline or ether, but this is the exception rather than the rule.

Note III.—Mention is made of the presence of barium carbonate but it is little used in American paints. Should it be found with soluble sulphates a decomposition will take place on treatment with acid forming insoluble barium sulphate and complicating the analysis. Only a very complete analysis of the bases and acid radicals would give sufficient data to form the probable combinations.

A paste paint has recently been introduced containing about 10 per cent. barytes (blanc fixe) and 90 per cent. barium carbonate. The analysis of such a mixture is easily made by dissolving in dilute hydrochloric acid, filtering and washing with hot water. The residue after ignition and weighing represents the barium sulphate. To the filtrate dilute sulphuric acid is added in slight excess and the barium sulphate obtained in the usual way is calculated to barium carbonate by the factor .845.

Note IV.—Consult notes I, II and III in Scheme I.

CALCULATIONS.

When Gypsum is Absent.—If no gypsum is present, calculate the sulphuric acid (SO_3) to lead sulphate by factor 3.7875, and deduct from the total lead weighed as sulphate. Then if neither whiting or magnesium carbonate is present calculate carbonic acid to basic carbonate of lead by factor 8.8

This will usually satisfy all the lead and is checked by calculating the basic carbonate of lead to lead sulphate by factor 1.172, and subtracting the result from the total lead sulphate. From the remaining lead sulphate deduct that calculated from the sulphuric acid and should any yet remain it is figured to lead oxide (PbO) by using factor .735. (see note 2). If whiting and magnesium carbonate are present it will be necessary to first deduct the CO_2 combined with them by multiplying calcium carbonate by factor .44 and magnesium carbonate by factor .521.

When Gypsum is Present.—If gypsum be present, calculate the sulphuric acid (SO_3) in it by factor .564 and deduct from the total sulphuric acid, calculating any remaining to lead sulphate by factor 3.78. Now deduct lead sulphate from total lead sulphate and the remaining lead sulphate is calculated to basic carbonate of lead, or, if no carbonates are present, to lead oxide (PbO). If carbonate of lime and magnesium are present deduct the carbonic acid contained in them before combining with the lead.

The calcium extracted by the nitric acid-alcohol treatment and weighed as sulphate, is deducted from the total calcium found and the difference is the lime existing as gypsum. As it is usually fully hydrated it will be necessary to calculate the calcium sulphate (CaSO_4) to $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ by factor 1.26, in which form it exists. Zinc is weighed as oxide and exists as such in the sample.

4. * Analyses of the Solid Portion of “Ready-Mixed”
Paints. (Light Tints.)

White Lead.....	32.84	33.87	27.26	20.35	59.67	11.92	24.08	25.04	
Zinc Oxide.....	63.36	60.26	54.45	26.58	34.73	30.79	59.00	69.60	
Lead Sulphate.....	2.35	4.88	1.33	18.90		8.36	0.13	1.00	
Whiting.....			14.18	28.70		43.94		0.68	
China Clay.....					4.52		14.96		
Barytes.....									
Insoluble Matter (Silica). ..			1.15	0.99		2.73		0.52	
Color (by difference).....	1.45	0.99	1.63	4.48	1.08	2.26	1.84	3.16	
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	
White Lead.....	42.60	16.63	8.84	23.70	5.45	17.97	35.83	21.41	
Zinc Oxide.....	49.96	33.41	64.15	44.55	33.70	43.00	44.11	58.00	
Lead Sulphate.....	3.10	4.45	0.58		19.38	3.13	0.53	1.00	
Whiting.....	0.57	6.90		29.03	17.76	3.48	2.96	2.15	
China Clay.....		7.56	10.09		5.95			13.50	
Barytes.....		25.84	14.27		13.44	32.42	12.73		
Insoluble Matter (Silica). ..	0.25			0.73					
Color (by difference).....	3.52	5.21	2.07	1.99	4.32		3.84	3.94	
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	

* By the Author.

5. * Analyses of White Paste Paints.

White Lead.....	22.73		24.10		28.95		32.24
Zinc Oxide.....	27.20		1.63		22.97		29.25
Lead Sulphate.....	36.78						19.31
Whiting.....	12.00						18.81
China Clay.....							
Barytes.....		74.08	72.80		45.80	53.23	
Insoluble Matter (Silica).....	0.99						
Lead Monoxide.....					1.43	8.58	
	99.70	99.76	98.53	99.15	99.33	99.61	
White Lead.....	21.86	23.12		32.54	23.86	52.72	
Zinc Oxide.....	24.11	4.20	55.44	33.64	4.70	8.68	
Lead Sulphate.....	33.60	57.76	0.18	0.16	50.26	32.54	
Whiting.....	7.62						
China Clay.....	11.33						
Barytes.....		14.44	44.36	33.17	20.58	5.93	
Insoluble Matter (Silica).....							
Lead Monoxide.....							
	98.57	99.52	99.98	99.51	99.40	99.87	

* By the Author.

APPENDIX.

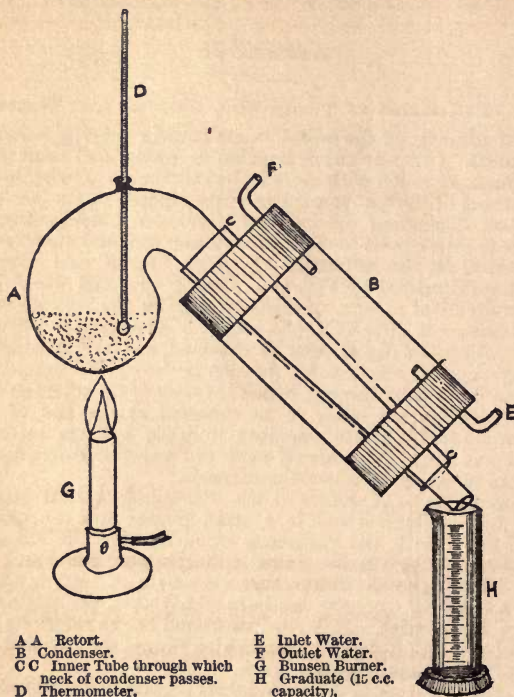
1. Estimation of Turpentine, Benzine and Water.

A majority of the mixed paints contain varying amounts of "turps," (turpentine) or benzine or water, and their estimation is attended with more or less difficulty. Owing to the proneness of "turps" to oxidize when exposed to the air any method depending on its distillation and condensation is liable to give results too low, as oxidation products are formed. A method for the estimation of water, "turps" and benzine has been proposed by Vernon J. Hall, Ph.D., and which has given excellent results, consists in mixing the sample with lead carbonate (PbCO_3) (previously dried in an air oven at 100°C.) until a dry powder is obtained, and then distilling, using a special condenser. As seen in the cut the condenser has an inner tube through which the neck of the retort passes, thus enabling the latter to be removed at the end of the operation if it becomes necessary to apply a flame to drive down any drop of condensed water or "turps" that frequently adhere to the inner tube of condensers.

From 20 to 25 grams of the *thoroughly* mixed sample are quickly weighed out in a small beaker and the beaker then filled with the carbonate of lead. By the aid of a spatula the whole is now mixed and chopped into fine powder. If it appears sticky more carbonate of lead is added and a sufficient quantity has been added when the mixture is a dry fine powder. It is now transferred to the retort by the aid of a sheet of glazed paper, folded funnel shaped, and any dust brushed down with a brush.

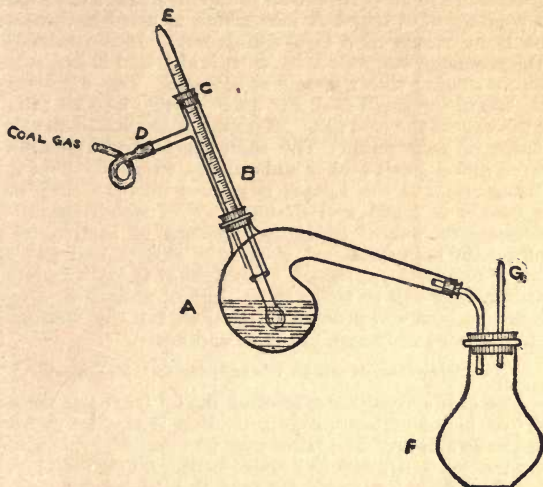
The graduate H is also weighed. Connect the apparatus and apply at first a low flame and gradually increase the heat until a temperature of about 120°C. is obtained which will be sufficient to volatilize all the water and benzine. Keep at the temperature until nothing more comes over. Now draw

the retort from the condenser and with the aid of the flame drive the remaining drops down into the graduate. The latter with its contents are weighed, and the increase of weight



equals water and benzine. If water is present it will be the lower layer and the volume of each is noted. Now increase the temperature to 170°C ., at which point all "turps" will

come over. When distillation ceases again remove the retort and with the flame drive down into the graduate any adhering drops. Read the volume. Any increase is due to turpentine. Weigh the graduate and contents, the increase in weight is total water, benzine and "turps." The percentages are found



as follows: Suppose 25 grams of sample were taken and the distillate weighed 2.1 grams, then

$$\frac{2.1 \times 100}{25} = 8.4 \text{ per cent. by weight of total distillation.}$$

Now the volumes read, benzine 1.2 c.c., water 1.5 c.c., "turps" 0.3 c.c., total 3.00 c.c., then

$$\frac{8.4 \times 1.2}{3} = 3.36 \text{ per cent. benzine by weight.}$$

Likewise, we would find 4.2 per cent. water and 8.4 per cent. "turps." Deducting this total distillate 11.2% from the

total mixing fluid as found in Chap. I, the difference is the percentage of oil by weight.

A method has been proposed by H. Joshua Phillip, F.I.C., F.C.S. (Chemical News, Vol. LXIII, No. 1646,) which consist of distilling in a stream of coal gas thus avoiding exposure to air. As shown in the illustration the apparatus consists of a retort A into which is fitted a three-way tube B by means of a tight fitting cork. In the tube B is a thermometer supported by a cork at C and D is a rubber tube connecting with a supply of coal gas. The distillate is collected in the small flask F. In F is fitted a small tube G for the exit of the coal gas. The flask F is allowed to rest in a basin of cold water. The thoroughly stirred sample is poured into a small beaker and quickly weighed. The tube B being removed the sample is poured into the flask until the beaker is quite well drained. Now weigh again, the difference in weight being the amount of sample taken. Connect the apparatus and start slow stream coal gas and allow to run for some time. Then light at G and regulate so that the flame will be about three-eighth of an inch in length. Now apply heat and proceed as detailed for the distillation and estimation of "turps," benzine and water.

Estimation of Oil in Flaxseed and Oil Cake.

The best solvent for extracting the oil from the flaxseed and cake is carbon bisulphide providing it can be obtained free from sulphur. The latter may be detected by evaporating a portion to dryness in a water bath. Should sulphur be present which is invariably the case it may be removed by carefully distilling off the carbon bisulphide. The grade that is made by Schnuchart (Germany) is very pure and contains such a slight trace that it need not be taken into consideration.

To estimate the oil; five grams of the oil cake are used or three grams of ground flaxseed and carefully brushed into a four ounce Erlenmeyer flask. Then add about 25 c.c. carbon bisulphide, cork loosely, and allow to stand about twelve hours with occasionally a gentle agitation. Pour the quite clear carbon bisulphide in a filter allowing it to run into a weighed beaker of about 200 c.c. capacity. Add about 15 c.c.

more bisulphide to the residue agitate gently and allow to stand about 15 minutes or until the sample is pretty well settled, again pour off and repeat the treatment about four times. Then with the aid of a wash bottle containing carbon bisulphide wash the remaining seed or meal to the filter, finish the washing of the beaker allowing the bisulphide to run on the filter and thoroughly wash the oil from the paper. When the carbon bisulphide is all run through place the beaker and contents in a warm place until the carbon bisulphide is evaporated then heat in an air oven at a temperature of 110° C. for about 15 minutes. Cool and weigh. The increase of weight equals the oil which multiplied by 100 and divided by the weight of sample taken gives the percentage.

2. List of the Principal Pigments with Chemical Formula and Trade Names.

WHITE PIGMENTS.

Chemical Name.	Formula.	Trade Name.
Sulphate of Barium...	BaSO_4	Barytes, "Blanc Fixe."
Carbonate of Calcium.	CaCO_3	Whiting, Lime Carbonate Lime, Paris White, English White, Spanish White, Marble Dust.
Sulphate of Calcium.	$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	Gypsum, Terra Alba, Plaster of Paris ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, when dehydrated.)
Sulphate of Strontium.	SrSO_4	Strontian White.
Sulphate of Lead.....	PbSO_4	Sublimed Lead, "White" Lead.
Silica.....	SiO_2	Silex, Silver White, Wood Filler, Infusorial Earth, Ground Quartz.
Oxide of Zinc.....	ZnO	Zinc White, Zinc Oxide, Zinc.
Hydrated Silicate of 2 Alumina.	$2 \text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$	China Clay, Clay.
Carbonate of Magn'm.	MgCO_3	Magnesite.
Silicate of Magnesium.	46% to 58% SiO_2	Talc, Soapstone, French
	29% " 34% MgO	Talc, Steatite.
	02% " 07% H_2O	
	(with small amounts Iron Oxide and Alumina.)	
Sulphide of Zinc.....	ZnS	Mixed with Barytes and Zinc Oxide is "Lithophone."
Basic Carb'te of Lead.	$2\text{PbCO}_3 \cdot \text{PbH}_2\text{O}_2$	White L'd, Corroded L'd.

GREENS.

Pure Chrome Green...	Cr_2O_3	Chrome Greens.
Chrome Green.....	PbCrO_4	Chrome Greens
	PbSO_4	
	$2\text{PbCO}_3 + \text{PbH}_2\text{O}_2$	
	$\text{K}_2\text{Fe}_2(\text{FeCN})_6$	
Chrome Green.....	$\text{K}_2\text{Fe}_2(\text{FeCN})_2$	Brunswick Green.
	PbCrO_4	
	PbSO_4	
	BaSO_4	
"Scheele's" Green.....	$\text{CuAsO}_3 \cdot \text{CuO} \cdot 2\text{H}_2\text{O}$	
"Emerald" Green.....	$7\text{Cu}_2\text{C}_2\text{H}_3\text{O}_3 \cdot 3\text{CuAs}_2\text{O}_4$	

BLUES.		
Chemical Name.	Formula.	Trade Name.
Ultramarine	Composition by Gmelin	
	SiO ₂	47.306
	Al ₂ O ₃	22.000
	Na ₂ O	12.063
	S	0.188
	CaCO ₃	
	CaO	1.546
	H ₂ SO ₄	4.679
	Water, etc...	12.218
Prussian Blue	Potassium Ferric Ferrocyanide K ₂ Fe ₂ 2Fe(CN) ₆	Berlin Blue, Chinese Blue.
REDS.		
Lead Monoxide	PbO	Litharge, Lead Oxide, Massicot.
Lead Oxide	Pb ₂ O ₄	Red Lead, Orange Red.
Basic Chromate of L'd.	PbCrO ₄ +PbO	Dark or Basic Chromate of Lead, Chrome Red, Scarlet Lead, Derby Red.
Sulphide of Mercury..	HgS	Mercury Vermilion, Chinese Vermilion.
Red Lead or Orange Mineral.		Artificial Vermilion, American Vermilion, Vermilionettes, Aniline Reds, Royal Reds.
Barytes as a base tinted with eosine (aniline).		
Oxide of Iron	Fe ₂ O ₃	Indian Reds, Rouge, Venetian Red, Princes Mineral, Tuscan Red.
Carbonate of Iron....	FeCO ₃	Crocus.
YELLOW AND ORANGE.		
Chromate of Lead....	PbCrO ₄ +PbSO ₄	Chrome Yellow, Chrome, (Sometimes white lead) Lemon Chrome.
Chromate of Lead....	PbCrO ₄	Orange Chrome.
Ochres and Siennas:—Silicate of alumina colored by hydrated ferric oxide in ochres and hydrated ferric oxide with manganese oxide in Siennas.		
BLACK PIGMENTS.		
Carbon	C	Lamp Black, Gas Black.
Carbon, with Phosphate of Calcium.	60% Phosphate of Calcium. 15% Carbonate of Calcium and some silica, iron and alumina. 25% Carbon	Ivory Black, Bone Black. Drop Black, Animal Black.
Graphite	C	Graphite.
BROWN PIGMENTS.		
Umber, both raw and "burnt," is the chief brown pigment. Essentially ochres or siennas but contain more manganese, the latter varying from 7 to 12 per cent. MnO ₂ .		

3. Atomic Weights.

Name.	Sym- bol.	Quanti- valence.	Atomic Weight.	Name.	Sym- bol.	Quanti- valence.	Atomic Weight.
Aluminium...	Al	IV	27.0	Mercury.....	Hg	II	200.0
Antimony.....	Sb	V	120.0	Molybdenum..	Mo	VI	96.0
Arsenic.....	As	V	74.9	Nickel.....	Ni	VI	59.0
Barium.....	Ba	II	136.80	Nitrogen.....	N	V	14.0
Bismuth.....	Bi	V	210.0	Osmium.....	Os	IV	199.0
Boron.....	B	III	11.0	Oxygen.....	O	II	16.0
Bromine.....	Br	I	80.0	Palladium....	Pd	IV	106.0
Cadmium.....	Cd	II	112.0	Phosphorus...P	P	V	31.0
Cæsium.....	Cs	I	133.0	Platinum.....	Pt	IV	197.0
Calcium.....	Ca	II	40.0	Potassium....	K	I	39.1
Carbon.....	C	IV	12.0	Rhodium.....	Ro	IV	104.0
Cerium.....	Ce	III	141.2	Rubidium.....	Rb	I	85.0
Chlorine.....	Cl	I	35.5	Ruthenium...Ru	Ru	IV	104.0
Chromium....	Cr	VI	52.4	Selenium.....	Se	II	79.0
Cobalt.....	Co	VI	59.0	Silicon.....	Si	IV	28.0
Columbium...Cb	Cb	V	94.0	Silver.....	Ag	I	108.0
Copper.....	Cu	II	63.1	Sodium.....	Na	I	23.0
Didymium....	D	III	147.0	Strontium....	Sr	II	87.5
Erbium.....	E	III	169.0	Sulphur.....	S	II	32.0
Fluorine.....	F	I	19.0	Tantalum....	Ta	V	182.0
Gallium.....	Ga	III	69.9	Tellurium....	Te	II	128.0
Glucinum....	Gl	II	9.2	Thallium....	Tl	I	204.0
Gold.....	Au	III	196.2	Thorium.....	Th	IV	231.5
Hydrogen....	H	I	1.0	Tin.....	Sn	IV	118.0
Indium.....	In	III	113.4	Titanium.....	Ti	IV	50.0
Iodine.....	I	I	126.85	Tungsten.....	W	IV-VI	184.0
Iron.....	Fe	VI	56.0	Uranium.....	U	VI	240.0
Lanthanum...La	La	III	139.0	Vanadium....	V	V	51.2
Lead.....	Pb	II	207.0	Yttrium.....	Y	III	60.0
Lithium.....	Li	I	7.0	Zinc.....	Zn	II	65.0
Magnesium....	Mg	II	24.0	Zirconium....	Zr	IV	90.0
Manganese....	Mn	VI	55.0				

4. Measures and Weights.

MEASURES OF CAPACITY.

Gals	Qts.	Pts.	Fl. Oz.	Fl. Dr.	Grains of Water at 62° F.	Cubic Centimetres.
1 =	4 =	8 =	128 =	1,024 =	58,318.00 =	3,785,200
	1 =	2 =	32 =	256 =	14,579.50 =	946,300
		1 =	16 =	128 =	7,289.75 =	473,150
			1 =	8 =	455.61 =	29,570
				1 =	56.98 =	3,690
1 English imperial gallon =				277.274 cu. in. =	70,000.00 =	4,543,000
1 " wine or Winchester gal. =			231.000	" =	58,318.00 =	3,785,200
1 " corn gallon =			268.000	" =	67,861.00 =	4,402,900
1 " ale " =			282.000	" =	71,193.40 =	4,619,200
		1	cu. ft. =	283.15 cc.		
		1	cu. in. =	16.38 "		
		0.061027	" =	1 "		

LINEAR MEASURES.

1 yd =	3 ft. =	36 in. =	0.91438 metre.
	1 ft. =	12 in. =	0.30480 "
		1 in. =	0.02540 "
		39.3708 in. =	1.00000 "

TROY WEIGHT.

1 lb. =	12 oz. =	240 cwt. =	5,760 grs. =	373.2419 grammes.
	1 " =	20 " =	480 " =	31.1035 "
		1 " =	24 " =	1.5552 "
			1 " =	0.0648 "

AVOIRDUPOIS WEIGHT.

1 gross ton	=	20 cwt.	=	2,240 lbs.	=	1,016.00 kilogrammes.
		1 "	=	112 "	=	50.80 "
				Oz.		Grs. Troy.
		1 lb.	=	16	=	7,000.00
				1	=	437.50
						Grammes.
						453.5926
						28.3495
1 net ton	=	2,000 lbs.	=			907 kilogrammes.
1 cu. ft. of water at 62° F.	=			62.3550 lbs. Av.	=	28,315.0000 grammes.
1 cu. in. "	"	"	"	0.0361 "	"	16.3862 "

APOTHECARIES WEIGHT.

1 lb. =	12 oz. =	96 dr. =	288 scruples =	5,760 grains =	373.2419 grams.
	1 " =	8 " =	24	480	31.1035 "
		1 " =	3	60	3.8879 "
			1	20	1.2960 "
0.0022 lb. Av. =	0.03527 oz. Av. =			15.4328 =	1.0000 "

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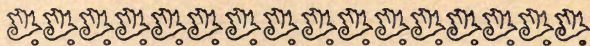
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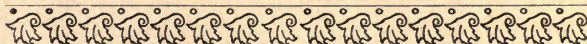
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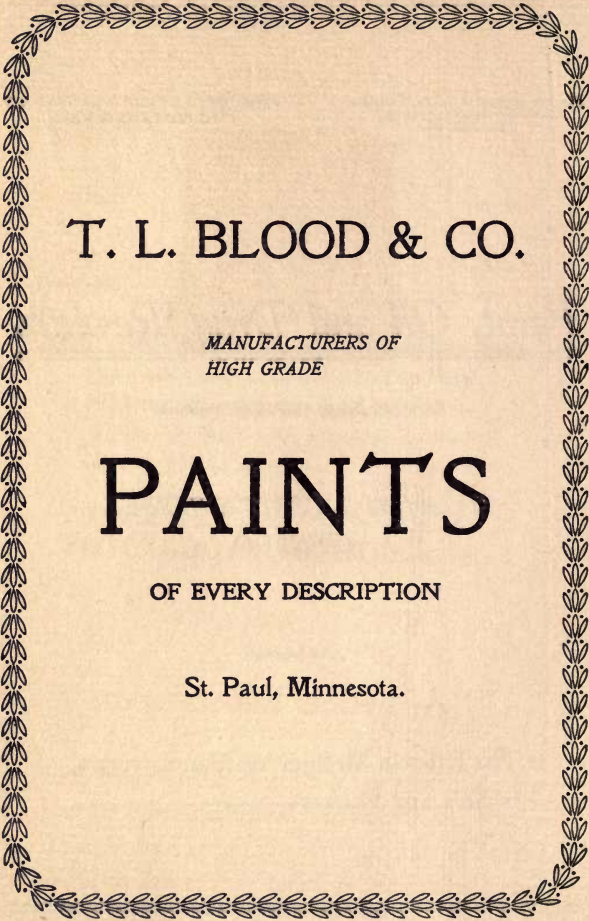
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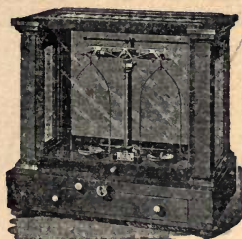
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